PB187770

DEVELOPMENT OF A SIMPLE AUTO EXHAUST ANALYZER

Prepared for:

DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
BUREAU OF STATE SERVICES, EH
PUBLIC HEALTH SERVICE
CINCINNATI, OHIO

ATTN: DR. BERNARD STEIGERWALD
LABORATORY OF ENGINEERING AND PHYSICAL SCIENCES
DIVISION OF AIR POLLUTION, SEC

CONTRACT PH 86-66-60

STANFORD RESEARCH INSTITUTE

MENLO PARK, CALIFORNIA

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MENLO PARK CALIFORNIA

August, 1966

Final Report | *Covering the Period January 24, 1966 to July 24, 1966*

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SRI Project FMU-5859

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PHYSICAL & INDUSTRIAL SCIENCES

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I INTRODUCTION

Air pollution by auto exhaust emissions has reached major proportions in many urban areas of the United States; control of such emissions is a public health problem urgently requiring alleviation. Some states already require crankcase and exhaust control devices on automobiles, and in all probability their use will be widely extended in the future. When control devices on automobiles are commonplace, simple analytical instruments will be required to maintain and check their performance.

Two "components" of auto exhaust for which maximum allowable concentrations have been spelled out in legislation are carbon monoxide and hydrocarbons. Stanford Research Institute, in a proposal to the Public Health Service Division of Air Pollution, outlined several approaches to a simplified exhaust analyzer that would measure total hydrocarbons and carbon monoxide separately. It was decided that the hot wire combustible gas detector principle had the best potential for these analyses. There were indications in the literature that, on the basis of filament temperature, one might separate the filament response due to total hydrocarbons from that due to carbon monoxide.

Under contract from the Public Health Service, Stanford Research Institute conducted a feasibility study to determine if the hot wire combustion principle could be applied to these analyses. This technical report describes the results of the study.

II SUMMARY

The aim of the research described in this report was to determine the feasibility of applying a heated filament combustible gas detector to the more demanding job of analyzing separately for total hydrocarbons and carbon monoxide in auto exhaust gases. The approach was based on the premise that the "components" might be resolved on the basis of filament operating temperature.

The first phase of the experimental work was a detailed study of the filament temperatures required for oxidation of selected hydrocarbons and carbon monoxide on a platinum filament. Additional work was done on the effect of oxygen and hydrocarbon concentration on oxidation initiation temperature, and the interaction effects in simple gas mixtures were studied. The data from these studies showed clearly that the required analyses could not be made on the basis of the platinum filament temperature.

A search was made for other materials which might show greater selectivity in catalyzing oxidation of hydrocarbons and carbon monoxide. The materials tested were rhodium, iridium, palladium, palladium-silver and palladium-gold alloys, oxidized nickel, gold and oxidized cobalt. None of these materials would make the required analyses feasible on the basis of filament operating temperature.

Two procedures were evaluated for making a preliminary separation before analysis with a heated filament. In the first of these, Hopcalite catalyst was used to remove the carbon monoxide from the gas sample by selective oxidation. While the oxidation could be made selective for carbon monoxide, problems associated with adsorption of hydrocarbons on Hopcalite made the procedure of doubtful use. In the second procedure, silica gel was used to selectively remove hydrocarbons from the gas sample so that an analysis for carbon monoxide could be made on the heated filament. While the method shows some potential

for making the required analyses, the complications it introduces are such that the basic simplicity of the combustible gas detector is lost.

Since the separate analyses for hydrocarbons and carbon monoxide did not seem feasible, an effort was made to construct a detector that would measure total combustibles minus hydrogen and the lower aliphatic hydrocarbons. The procedure utilized a platinum wire "preburner" whose function was to selectively combust hydrogen. The gas stream would then be analyzed over a palladium filament which, according to our previous work, would respond to carbon monoxide, olefins, and acetylenics but would not respond to aliphatics at least up to butane. This procedure did not succeed because carbon monoxide was found to inhibit the combustion of hydrogen over platinum at low temperatures to such an extent that the selective combustion of hydrogen could not be accomplished.

Our conclusion, based on the work described here, is that the combustible gas detector does not show any great potential for auto exhaust analysis.

III EXPERIMENTAL WORK

A. Apparatus

The sample preparation system used in this work consisted of an 8 liter stainless steel tank connected to a manifold fitted with a vacuum pump, Zimmerli type vacuum gage, purified compressed air supply, and gas sample introduction ports. Test mixtures were prepared by pumping the stainless steel tank down to a few millimeters residual pressure, introducing appropriate pressures of sample gases, and then pressurizing the sample tank to 85 psig with purified compressed air. The prepared mixture was then metered through the detector cell, using needle valves for flow control and an orifice flow meter for measuring flow rates.

A breadboard model combustible gas detector was constructed using commercially available platinum filaments.* All of the experimental work was done using a dual filament arrangement with the active filament in the sample gas stream and the reference filament in still air. The other half of the detector bridge was a combination of two 220 ohm fixed resistors and a 25 ohm Helipot for zero control. The bridge power was supplied by a Quan-Tech Model 101-1 voltage-regulated adjustable power supply. Bridge current was monitored by a 0-2 amp ammeter. The bridge output was recorded on a Varian G10 strip chart recorder. A calibrated 0-100 millivolt voltage reference source was placed in series with the recorder high input terminal so that a known suppression voltage could be introduced if the signals were too large for the recorder span.

B. Oxidation Experiments--Platinum Filaments

The initial experimental effort was concerned with a detailed study of the heterogeneous catalytic combustion of selected hydrocarbons and carbon monoxide over a heated platinum filament. The

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* Johnson Williams Co., 2300 Leghorn Street, Mountain View, California.

objective of this work was to determine the feasibility of making a separate analysis for total hydrocarbons and carbon monoxide on the basis of filament temperature. To this end, the voltage-current characteristics of a set of platinum filaments were carefully measured so that the resistance and consequently the temperature of the filaments as a function of filament current could be inferred. The temperatures arrived at by this procedure are average values since temperature gradients of unknown magnitude exist over the length of the filament. Next the oxidation initiation temperatures were measured for a selected group of hydrocarbons and carbon monoxide. These data were obtained by flowing 20 cc/min of the gas sample over the platinum filament detector and gradually increasing the filament current (temperature) while monitoring the detector bridge output voltage with the strip chart recorder. The onset of oxidation was indicated by an abrupt change in bridge output signal. The filament current at which oxidation started was converted to temperature using the relations described above. Concentrations used for these experiments were all in the area of 3000 ppm (v/v). Table I lists the compounds studied and the filament temperatures at which oxidation was initiated.

Table I

OXIDATION INITIATION TEMPERATURES OVER PLATINUM

Compound	Oxidation Initiation Temperature, °C
Methane	625
Ethane	515
Propane	340
n-Butane	290
n-Pentane	210
n-Hexane	185
n-Heptane	165
n-Octane	170
Isoöctane	170
Cyclohexane	200
Ethylene	290
Propylene	280
Isobutene	240
Cyclohexene	300
Acetylene	500
Propyne	435
2-Methyl-1-butene-3-yne	>625
Benzene	265
Carbon Monoxide	290

The dependence of initiation temperature on concentration of hydrocarbon was determined; data for butane-air mixtures are presented in Table II.

Table II

CONCENTRATION DEPENDENCE OF INITIATION TEMPERATURES

Butane Concentration, ppm(v/v)	Oxidation Initiation Temperature, °C
9600	245
4800	255
2400	270
1200	300
600	325
300	360

A plot of $\log_{10}$ concentration versus reciprocal temperature is a straight line of unit slope.

Some experiments were done to determine if the initiation temperatures determined using one component at a time would be the same in simple mixtures of gases. A mixture of about 1000 ppm each of methane and butane was passed over the active filament while its temperature was systematically increased. The bridge output signal was noted for each filament current value. When the bridge output was plotted against the filament temperature, a curve with two plateau regions was obtained. For these components, whose initiation temperatures are about 300°C apart, no significant interaction effects occur. Similar experiments were done using butane and acetylene (initiation temperatures about 200°C apart). A plot of these data also showed two plateau regions. One other pair, carbon monoxide-methane, was examined using this technique. This pair was easily resolved on the basis of platinum filament temperature.

A series of measurements was made to determine the effect of oxygen concentration on initiation temperatures and signal magnitude using butane at 3500 ppm in air and air-nitrogen mixtures. A butane-air-nitrogen mixture was prepared with just twice the stoichiometric amount of oxygen for complete oxidation of butane to CO_2 and H_2O. This mixture was compared to a butane-air mixture having ten times the stoichiometric amount of oxygen. From measurements on these mixtures it was concluded that a low oxygen concentration decreases the initiation temperature from 265°C to 235°C and the signal magnitude (filament temperature rise) increases slightly.

Consideration of the data on initiation temperature over platinum filaments leads to the inescapable conclusion that a differentiation of total hydrocarbons, as a class, from carbon monoxide on the basis of filament temperature alone is not feasible. Many of the most "reactive" hydrocarbons combust over heated platinum in the same temperature region as carbon monoxide. On the basis of this conclusion the laboratory effort was realigned in the direction of searching for metals with more specific catalytic activity, and to investigate some simple separation procedures that would still retain the basic simplicity of the heated filament combustion analysis.

C. Platinum Family Metals

The following platinum family metals and alloys were examined for their ability to catalyze selectively the combustion of hydrocarbons and carbon monoxide:

Rhodium

Iridium

Palladium

Palladium-Silver (85%-15%, 70%-30%, 40%-60%)

Palladium-Gold (60%-40%, 25%-75%)

The behavior of each of these is described below.

Rhodium filaments are not catalytically active for combusion of carbon monoxide and hydrocarbons until they have been heated in air to around 600°C. At this temperature a surface oxidation of the rhodium takes place, and the filaments are then very active. The first indications, when oxidized rhodium filaments were used for oxidation initiation tests, were favorable, in that carbon monoxide oxidation initiated at a significantly lower temperature than did pentane oxidation. However, continued work soon revealed a problem; the active catalyst (presumed to be rhodium oxide) was not stable when heated in the presence of carbon monoxide.

A systematic study of the carbon monoxide initiation current (temperature-current relationship for this material was not determined) was made in the following way: the rhodium filament was freshly oxidized in air and the carbon monoxide initiation current was determined. The filament was then used to oxidize the same carbon monoxide-air mixture at a current somewhat above the original initiation current for a fixed gas flow and time. The initiation current for this mixture was determined again. This process was repeated several times for increasing filament currents. The results of these tests show that the activity of the filament, as indicated by signal response and initiation current, declines as it is used to oxidize carbon monoxide. The decline is most pronounced when the carbon monoxide is oxidized at currents close to the original initiation current. For example, a

freshly oxidized filament initiates carbon monoxide oxidation (5000 ppm) at 0.47 amp. After oxidizing this mixture for 20 minutes at 0.55 amp (flow rate 30 cc/min), the initiation current increases to 0.53 amp. This represents an increase in initiation temperature on the order of 50°C. If the carbon monoxide-air mixture is oxidized for 20 minutes at 0.65 amp, the initiation current increases to only 0.49 amp; thus it is possible to retain the catalytic activity of oxidized rhodium filaments but only by operating at a temperature where carbon monoxide and hydrocarbons will oxidize together. Similar tests were made for a butane-air mixture; no decline in filament activity was produced as a result of hydrocarbon oxidation. Based on these results one can conclude that rhodium filaments do not meet the requirements for a simplified auto exhaust analyzer.

Some observations on wire filaments of iridium were made. Its behavior was similar to that of rhodium in oxide formation and degradation, but its ability to discriminate carbon monoxide from hydrocarbons was inferior to that of rhodium.

Palladium filaments gave encouraging preliminary results in that carbon monoxide oxidized at quite low temperatures, and butane could not be oxidized at all; further tests with ethylene and acetylene demonstrated that they oxidize at nominally the same temperature as carbon monoxide. The activity of palladium toward other aliphatics was not determined, since it could not discriminate major components of auto exhaust like acetylene and ethylene from carbon monoxide.

Some brief tests on palladium-silver alloys (70% Pd - 30% Ag) were carried out. This material has shown some interesting properties in other catalyst studies at the Institute. It is nominally inactive for oxidation of hydrocarbons and carbon monoxide.

Two palladium-gold alloys (60% Pd - 40% Au and 25% Pd - 75% Au) were also tested. These were active for carbon monoxide and hydrocarbon oxidation, but with no special selectivity. The activity is proportional to the palladium content.

D. Nonplatinum Metals and Oxides

A set of resistance-matched nickel wire filaments was constructed and evaluated. The filaments were helically wound from 2-1/2 inches of 0.005-inch-diameter wire and, after mounting on a threaded support bushing, they were surface-oxidized by electrically heating in air. In a screening test the filaments were checked for response to butane, carbon monoxide, ethylene, and acetylene. The result was that oxidized nickel wire seems to be catalytic only for oxidation of acetylene; the oxidation is initiated in the range of 300-400°C. No instrument response was observed for butane, carbon monoxide, or ethylene until the filament temperature was around 700-900°C. At these temperatures a very minor response was observed; however, this is most likely due to a homogeneous (gas phase) reaction that would be initiated by any surface at such a high temperature.

A set of gold wire filaments was constructed and tested for response to pentane and ethylene. Gold was catalytically inactive in these tests.

Because several research papers have noted the activity of oxides of cobalt as catalysts for carbon monoxide and hydrocarbon oxidation, cobalt wire was ordered for evaluation work. A preliminary test of cobalt oxide activity was made by fabricating a set of filaments in the following way. A 0.005-inch-diameter nickel wire was formed as a helical coil and the wire was superficially oxidized by electrically heating in air. The nickel was oxidized to form a surface to which the subsequently produced cobalt oxide would adhere. The oxidized filament was then treated with aqueous cobalt nitrate solution, dried, and heated electrically to decompose the cobalt nitrate to cobalt oxide. The exact nature of the oxide formed is not known; however, this procedure produced a very active catalytic filament which seemed to have some ability to differentiate carbon monoxide from butane on the basis of temperature. Further work on these filaments, however, demonstrated that they lack stability. A decline in catalytic activity, similar to that experienced with rhodium, occurs when they are used for oxidizing

carbon monoxide. The sensitivity (signal output) of the filament as a function of filament temperature exhibits a sharp maximum when oxidizing carbon monoxide; this factor would make an analysis based on filament temperature unreliable.

Some work was done on filaments made of pure cobalt wire. When heated to a temperature at which cobalt oxide formed and the filaments were catalytically active, the resistance of the filaments continually changed with time. This drift is probably due to the nonprotective nature of the oxide formed.

E. Preliminary Separation Procedures--Hopcalite Selective Oxidation

Since no catalytic filament was found that would allow separate analysis for hydrocarbons and carbon monoxide based on filament temperature, other approaches based on selective removal of one of the "components" were investigated. The first of these involved using Hopcalite, a proprietary catalyst of Mine Safety Appliances Co., to selectively oxidize carbon monoxide, after which one could measure the residual hydrocarbons. Another measurement on the whole gas sample would allow analysis for carbon monoxide by difference.

The Hopcalite was contained in a glass tube with a bypass arrangement just upstream from the platinum filament detector. Its efficiency in converting carbon monoxide to carbon dioxide was evaluated at room temperature, 60°C, and 125°C by comparing the signal from the hot filament detector when the gas mixture was passed through the Hopcalite with that when it was bypassed around the Hopcalite. Some oxidation efficiency observations were also made by bubbling the detector cell exit gas through a saturated calcium hydroxide solution and estimating the quantity of calcium carbonate produced. While the Hopcalite effectively oxidizes carbon monoxide at all three temperatures used, a test hydrocarbon, butane, was adsorbed very strongly on the Hopcalite at the lower temperatures and partially oxidized at the higher temperatures.

An effort was made to optimize the procedure by reducing the quantity of Hopcalite used and finding optimum temperatures where oxidation of carbon monoxide would be retained and adsorption of hydrocarbons would be minimized. A 6-cm long by 0.4-cm I.D. packed Hopcalite tube was constructed with a thermocouple inserted in the center of the packing. The efficiency of this column, operated at 67°C and a flow rate of 30 cc/min, was adequate for reducing realistic carbon monoxide concentrations to zero. A series of tests was made to determine retention time and recovery efficiency for butane at various temperatures between 30°C and 108°C. The recovery was estimated on the basis of the fraction of the signal obtained when the butane-air mixture passed through the Hopcalite compared to the signal when the sample bypassed Hopcalite tube. Up to 57°C essentially all the butane is recovered, but the time for signal to reach its maximum value when the gas stream is switched to the Hopcalite tube is about nine minutes. At higher temperatures the recovery of butane is less (as little as 45% at 87°C) and retention times are relatively shorter. Several other hydrocarbons--acetylene, ethylene, hexane, and benzene--were tested at 57°C. The recoveries ranged from 50% to about 90% and retention times were longer than 30 minutes in the cases of hexane and benzene.

The rather poor recovery of hydrocarbons, coupled with the long retention times, suggest that this selective oxidation procedure would not be useful in the design of a simplified exhaust analyzer.

F. Silica Gel--Selective Adsorption of Hydrocarbons

The second approach to making a preliminary separation of exhaust "components" involved the use of a silica gel adsorbent column. The procedure was to make a measurement on the whole gas stream and then shunt the gas stream through a silica gel column on which the hydrocarbons would be retained for a sufficient time to allow a measurement of carbon monoxide, for which retention time in the column is very short. This approach was tested using a 3-foot by 1/4-inch column packed with Davison Grade 12 silica gel, and operated at room temperature.

Retention times (based on the time when the material first appeared at the detector) for several materials compared under a standard set of operating conditions were:

Compound	Retention Time, minutes
Carbon Monoxide	< 1
Methane	2
Ethane	11
Ethylene	30
Acetylene	> 78

The only hydrocarbon that could interfere in this procedure is methane, and in the report section on combustion over a platinum filament it was reported that carbon monoxide and methane can be distinguished on the basis of filament temperature. This procedure, while it has some potential for allowing separate measurements for carbon monoxide and hydrocarbons, is considered too cumbersome to be applied to a simplified exhaust analyzer. In addition, the interference problem presented by hydrogen, a major component in exhaust, would still have to be solved.

G. "Total Combustibles" Analyzer

Near the end of the contract period, and upon review of all the prior experimental data, it was jointly decided (by SRI staff and Dr. Steigerwald of the Public Health Service) that the remaining time should be used in attempting to demonstrate the feasibility of making a combustible gas analyzer that would measure total combustibles minus hydrogen, and the lower aliphatic hydrocarbons. Our previous work on oxidation initiation temperatures over platinum suggested that one might selectively combust the hydrogen in a mixture of hydrogen, carbon monoxide, and hydrocarbons by passing the gas mixture over a platinum "preburner" operated at a fairly low temperature. Aliphatics, at least up to butane, can be rejected by using palladium for the analysis filament.

A "preburner" was constructed in the shape of a 5-inch long, 0.1-inch diameter helical platinum coil mounted in a close-fitting, thick-wall, glass tube. The preburner coil was heated by passing current through it. Operated at 105°C, this preburner was able to reduce a 1000-ppm concentration of hydrogen to nominally zero. At this temperature, carbon monoxide came through the preburner intact. However, when a mixture of hydrogen and carbon monoxide was passed over the preburner filament, the hydrogen no longer combusted. Upon raising the preburner temperature, one could get combustion but it was not selective; that is, both hydrogen and carbon monoxide burned. Carbon monoxide thus inhibits hydrogen combustion over platinum at low temperatures.

At this point almost all the candidate filament materials previously examined were checked for selectivity with respect to hydrogen combustion in the presence of carbon monoxide. None of these materials was capable of selective combustion of hydrogen, usually because of the closeness of initiation temperatures for hydrogen and carbon monoxide. The oxidation of hydrogen over palladium does not seem to be inhibited by carbon monoxide, but the initiation temperatures are almost the same. A filament was constructed from 80% Pt - 20% Pd wire; the purpose was to determine if a small amount of palladium in platinum might remove the inhibitory effect of carbon monoxide on platinum while retaining temperature selectivity of platinum for hydrogen oxidation. Tests of this filament suggested that the behavior was in the direction of better selectivity; however, the data are rather tenuous, and for lack of time, no further work was done.

IV DISCUSSION AND CONCLUSIONS

A laboratory evaluation has been completed regarding the feasibility of applying a heated filament, combustible gas detector to the separate measurement of total hydrocarbons and carbon monoxide in auto exhaust. At the outset it was recognized that many problems existed; among these were interference from hydrogen, poisoning of catalytic filaments by lead and sulfur compounds, interference from nitric oxide, problems from particulate matter, etc. Most of these problems did not enter into the program because no satisfactory way was found to accomplish the first step--the separate analyses for hydrocarbons and carbon monoxide.

Of the several candidate filament materials tested, only two--oxidized rhodium and wire-supported cobalt oxide--showed any potential for making the required analyses on the basis of temperature, and these both lacked the required stability to be useful.

Two procedures for making a preliminary separation of the components were evaluated: selective oxidation of carbon monoxide over Hopcalite, and selective adsorption of hydrocarbons on silica gel. Neither of these was appropriate for incorporation in a simplified exhaust analyzer.

The final experimental effort, a compromise on the original requirements, was an effort to construct a "total combustibles" analyzer. This approach was unsuccessful because no way was found to selectively combust hydrogen in the presence of carbon monoxide. It can only be concluded that the heated filament combustible gas detector has no promising application to auto exhaust analysis.

V LITERATURE SURVEY

During the early part of the program a literature survey on the following topics was made:

1. Exhaust gas composition and analysis
2. Heated filament, combustible gas detectors and reactions
3. Oxidation of hydrocarbons, carbon monoxide, and hydrogen
4. Catalysts for oxidation of hydrocarbons, carbon monoxide, and hydrogen

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Printed in U.S.A. August 1966